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SOLVENT EXTRACTION AND SPECTROPHOTOMETRIC DETERMINATION OF NI(II) BY USING 5-BROMO SALICYLIDENE-2-AMINOTHIOPHENOL AS AN ANALYTICAL REAGENT

SANJAY K. PATIL*

C. K. Thakur A.C.S. College, New Panvel, Raigad, Mumbai, Maharashtra, India

ABSTRACT

Solvent extraction and spectrophotometric determination of metal ions are most versatile methods used for the removal, separation and concentration of metallic species from aqueous media. A spectrophotometric method has been developed for the determination of Nickel, Ni(II) using 5-bromo salicylidene-2-aminothiophenol as novel extractive reagent. The reagent forms a coloured complex which has been quantitatively extracted into n-Butanol at pH 7.8. The method obeys Beer's law over a range of 1 to 10 ppm. The molar absorptivity is 7428 L $mol^{-1}cm^{-1}$ and Sandell's sensitivity is 0.02083 µg/cm^2 respectively. The proposed method is very sensitive and selective. This method has been successfully applied for the determination of nickel from various alloys, synthetic mixtures and vegetable oil samples.

KEYWORDS: *Solvent extraction, spectrophotometric determination, 5-bromo salicylidene-2-aminothiophenol, n-butanol, molar absorptivity, Sandell's sensitivity.*

Sanjay K. Patil*

C. K. Thakur A.C.S. College, New Panvel, Raigad, Mumbai, Maharashtra, India

***Corresponding Author**

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INTRODUCTION

Nickel(II) is an important and toxic metal ion that is readily encountered in the environment, biological samples and food.[1]In addition, industrial waste such as scrap, dust, sludge, waste water and alloy also contain nickel to a great extent.[2,3]The cursory look at the literature survey reveals the fact that nickel reacts with many organic reagents[4,5]. Solvent extraction, which is one of the most versatile methods used for the removal, separation and concentration of metallic species from aqueous media.[6] It has been used widely for the extraction of Ni(II) ions.[7] Extraction systems containing amines and their salts are widely used to concentrate and separate metals. From a theoretical viewpoint, these systems are among the most complex because of the variety of intermolecular interactions that can occur in the organic phase. Studies of the extraction of metals using long chain amines have shown that Ni is extracted to a negligible extent from HCl solutions[8,9].In the present study, a novel method has been developed using 5-bromo salicylidene-2-aminothiophenolas a reagent for extraction and spectrophotometric determination of Ni(II).This method involves systematic monitoring of the influence of several process parameters: the pH of aqueous phase, the concentration of extractant, the extraction time, stability of the complex and nature of the extracted species. This new method of extraction of Ni(II) ions using 5-bromo salicylidene-2-aminothiophenol(BSATP) as extractant is simple, selective and sensitive.

EXPERIMENTAL

Reagents

Nickel sulphate, n-butanol, 8-hydroxy quinolone, thiourea, various solvent used and other required chemicals were purchased from S.D. Fine Chemical Ltd., India.

Preparation of reagent

The reagent 5-Bromosalicylidine-2-aminothiophenol was synthesized by following method.The 5-Bromo salicylaldehyde (0.01 mol) was taken in a round bottom flask and dissolved in the ethanol (20 ml). The alcoholic solution of 2-aminothiophenol (0.01 mol) was added slowly with constant stirring in round bottom flask. The resulting mixture was refluxed on a water bath for 4 hours. After cooling at room temperature or on concentrating the reaction mixture by partial solvent removal, solid Schiffs base 5-Bromosalicylidine-2-aminothiophenol separated out. It is then filtered at suction pump and washed thoroughly with dilute methanol followed by distilled water. The reagent was recrystallized from ethanol.

Preparation of stock solution

The stock solution of Ni(II) was prepared by dissolving a weighed amount of nickel sulphate in double distilled water and then diluted to the desired volume with double distilled water. An aliquot of this solution was used for gravimetric determination of nickel by dimethyl glyoxime method.[10]

Apparatus and measurement

A Shimadzu UV-Visible 2100 spectrophotometer was used to measure Nickel(II) concentration in1 cm cells with detection at 445nm. The pH of aqueous solution was measured using a digital pH meter with combined glass electrode.

Procedure for the extraction

To the 1 ml of aqueous solution having 0.1mg of nickel metal in a 50 ml beaker, 2 ml of 0.05% reagent was added. The pH of the solution attuned to 7.8. The total volume should not surpass 10 ml. The solution was filled in 100 ml separatory funnel. The beaker was washed twice with CHC_{l3} and washings are collected in the same separatory funnel. This separatory funnel was shaken for 1 minute and set aside to allow the two phases separate out. Anhy.Na_2SO_4 is added for eliminating the traces of water from organic phase. It is then collected in 10 ml measuring flask and diluted upto the mark with organic solvent if necessary. The spectrophotometric method is employed to estimate the amount of nickel present in the organic phase by measuring absorbance at 445 nm. The dimethyl glyoxime method was used to determine the amount of nickel in aqueous phases.[10]

RESULTS AND DISCUSSIONS

The results of various studies are discussed below.

Extraction as a function of pH

The extraction of nickel with5-bromosalicylidene-2-aminothiophenol has been studied over the pH range 2 to 10 and has been observed that percentage extraction of Ni (II) is maximum at pH 7.8.(Fig. 1). In previous work Ni(II) was extracted at acidic pH of 4.5.[7]

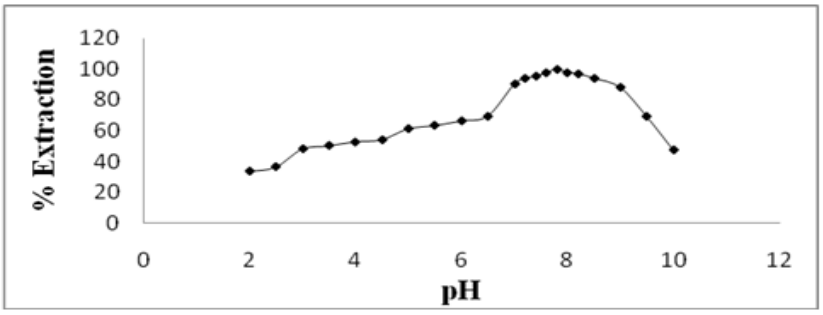

Figure 1
Effect of pH on % extraction

Absorption spectrum

The absorption spectrum of Ni(II): 5-bromo salicylidene-2-aminothiophenol in n-butanol shows the maximum absorption at 445 nm. The absorption due to reagent at this wavelength is nearly negligible. Hence the absorption measurements were carried out at 445nm. (Fig. 2)

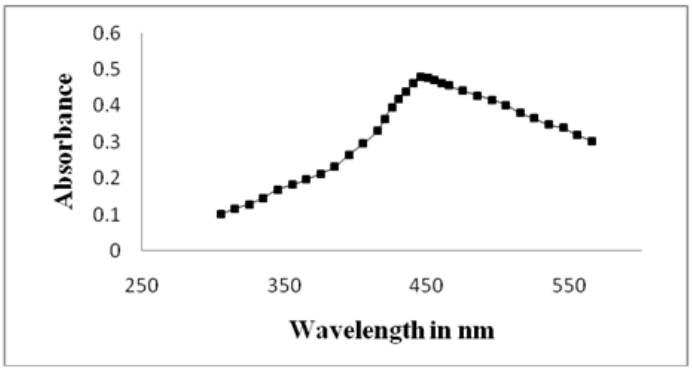

Figure 2
Absorption Spectrum

Influence of diluents

The suitability of solvent was investigated using various organic solvents and the extraction of Ni(II) was quantitative in n-Butanol. Hence, n-Butanol was used for further extraction studies as it gave better and quicker phases separation[12]. (Fig. 3)

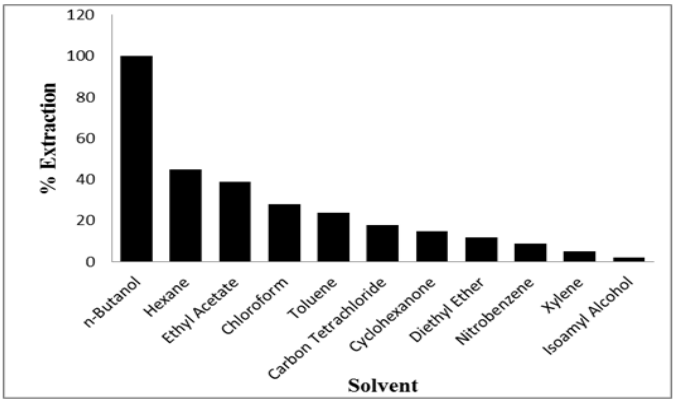

Figure 3
Effect of various solvents on Ni (II): BSATP complex

Effect of reagent concentration

It was found that 2 ml of 0.05% reagent is sufficient for the colour development of the Ni(II):BSATP complex in 10 ml of aqueous solution at pH 7.8. (Fig. 4). In the previous work, high concentration of triethyl amine reagent (5%) was used to extract Ni(II).[7]

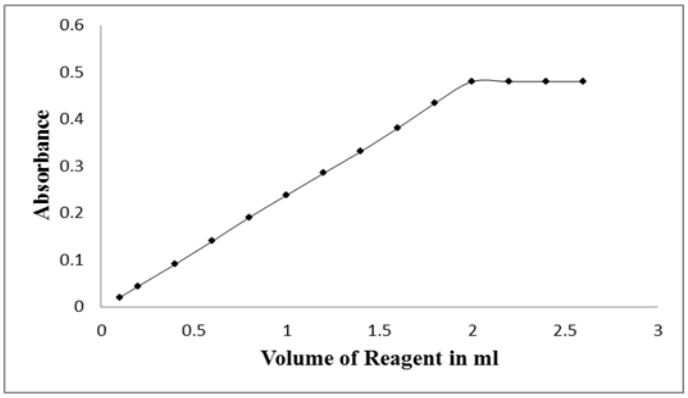

Figure 4
Effect of Reagent Concentration on Ni (II):BSATP complex

Effect of equilibration time and stability of the complex

The equilibration time 1 minute is sufficient for the quantitative extraction in n-Butanol. The stability of colour of the Ni(II):BSATP complex with respect to time shows that the absorbance due to extracted species is stable up to 60 minutes, after which slight decrease in absorbance is observed . (Fig. 5)

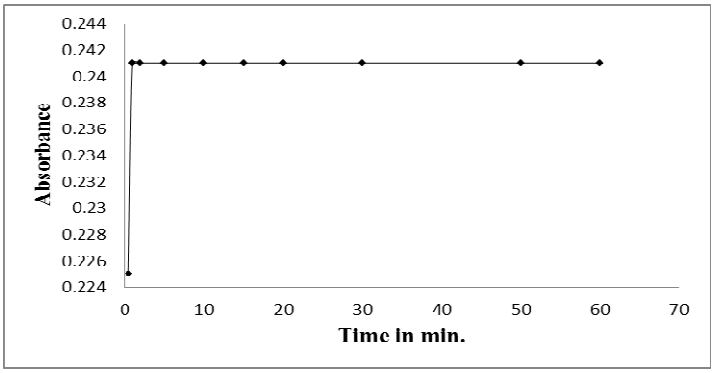

Figure 5
Effect of Time on Equilibrium on the Absorbance of Ni(II): BSATP complex

Calibration plot

The Beer's law is obeyed from 1 to 10 ppm. The molar absorptivity and Sandell's sensitivity were calculated to be 7428 L $mol^{-1}cm^{-1}$ and 0.02083 µg/cm^2 respectively. (Fig. 6)

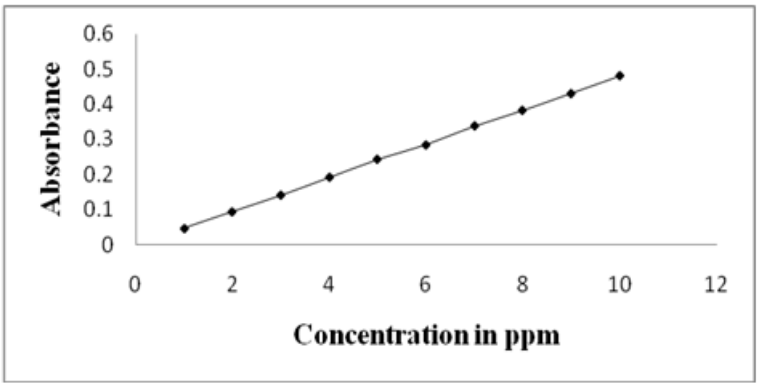

Figure 6
Calibration graph

Effect of divalent ions and foreign ions

The effect of other ions present in various amount indicated no interference in the spectrophotometric determination of 10 ppm of nickel. The ions which show interference in the spectrophotometric determination of nickel were overcome by using appropriate masking agents. [6, 11] (Table no.1)

Table 1

Sr. No.	Interfering Ions	Masking Agents
1	U^{6+}	8-Hydroxy Quinoline
2	Pd^{2+}	Thiourea
3	Citrate	Sodium Molybdate
4	Tartarate	Sodium Molybdate
5	EDTA	Sodium Molybdate
6	Pb^{2+}	Sodium Sulphate
7	Mo^{6+}	Citrate
8	Cu^{2+}	EDTA/Sodium Thiosulphate
10	Fe^{3+}	Alkali Cyanide/Thiourea
12	Cr^{2+}	Ammonium Acetate

Precision and accuracy

The precision and accuracy of the spectrophotometric method have been studied by analyzing five solutions each containing 5 ppm of nickel in the aqueous phase. The average of five determinations was 5.06 and variation from mean at 95% confidence limit was 5.06 ± 0.04482.

Sr. No.	Absorbance	Amount of Ni(II) (μg)	X– Mean	$D = X_i - X$	$D = X_i - X^2$
1	0.241	5		0.016	0.000256
2	0.246	5.08		0.008	0.000064
3	0.237	4.95	5.016	0.066	0.004356
4	0.241	5		0.016	0.000256
5	0.244	5.05		0.034	0.001156

Nature of extracted species

The composition of Ni(II):BSATP complex has been determined by Job's continuous method, Slope ratio method and Mole ratio method. It showed that the composition of Ni(II):BSATP complex is 1:2 (Fig. 4)

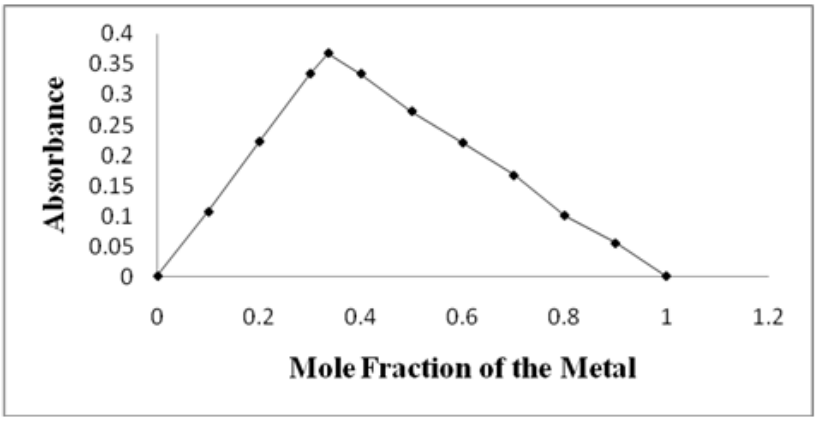

Figure 4
Job's continuous variation curve

Applications

The proposed method has been successfully applied for the determination of nickel from various alloys, synthetic mixtures and vegetable oil samples. The results found to be in good agreement with those obtained by the standard known method (Table No.2).

Table 2
Estimation of nickel in alloys, synthetic mixtures and vegetable oil samples

Estimation of nickel in	Sample	% Ni (II) certified value	% Ni (II) observed value	Standard Deviation
Alloys	Cupro-Nickel Alloy	55	54.8	± 0.2
	Magnesium Alloy	30	29.2	± 0.70
	German alloy	92	90.08	± 1.8
Synthetic mixture	Ni + Zn	4.91	5.0	± 0.09
	Ni + Co	4.88	5.0	± 0.10
	Ni + Mg	4.86	5.0	± 0.12
Vegetable oil	Groundnut oil	3.5mg	3.62mg	± 0.08 mg

Each result is average of three independent determinations.
Each result is compared with dimethyl glyoxime method.[10]

CONCLUSION

This study reveals that 5-bromo salicylidene-2-aminothiophenol BSATP can be used as an effective reagent for the extraction of Ni(II) ions from aqueous solutions. The extraction of Ni ions is pH-dependent and sensitive to the concentration of the extractant. The extracted species possessed a metal-to-reagent mole ratio of 1:2. After three extraction/stripping cycles, BSATP in n-Butanol retained its high extractability. The results show good agreement with standard method. The developed method is compared with results obtained with the dimethyl glyoxime method for estimation of nickel (II) and observed to be comparable. The method is precise, accurate, less time consuming and easily employed anywhere even in small laboratories as it requires only UV-visible spectrophotometer and not much sophisticated and costly measurement devices or instruments.

CONFLICT OF INTEREST

Conflict of interest declared none.

REFERENCES

1. Lucia Helena S. Avila Terra, Madalena C. da Cunha Areias, Ivanise Gaubeur, Maria Encarnación & V. Suárez-iha. Solvent Extraction-Spectrophotometric Determination of Nickel(II) in Natural Waters Using DI-2-Pyridyl Ketone Benzoyl hydrazone. Spectroscopy Letters.2008 April 17; 32(2):257-271.
2. Kim, T.Y., S.J., Kim, J.H. Yang, and S.Y. Cho. Environmentally Friendly Separation of Heavy Mental Ions onto Porous Chitosan Beads. J. Ind. Eng. Chem. 2004 March; 10(2):201-207
3. Choong Jeon, and Sung Ho Yeom. Treatment of Heavy Metals in Contaminated Soils Around an AbandonedMine Using Magnetically Modified Alginic Acid and XYZ. J. Ind. Eng. Chem.2006 May; 12(3):362-367.
4. Juan Costamagnaa, Luis E Lilloa, Betty Matsuhiroa, Miguel D Nosedab, Manuel Villagrána. Ni(II) complexes with Schiff bases derived from amino sugars. Carbohydrate Research. 2003 July 15; 338(15):1535-1542.
5. Md. Rabiul Hasan, Mohammad Amzad Hossain, Md. Abdus Salam, Mohammad Nasir Uddin. Nickel complexes of Schiff bases derived from mono/diketone with anthranilic acid: Synthesis, characterization and microbial evaluation. Journal of Taibah University for Science. 2016 September; 10(5):766-773.
6. R. S. Lokhande, S. P. Janwadkar, SantoshKulkarni and Sanjay Patil. Solvent extraction and spectrophotometric determination of cu(ii) by using acetophenone 2',5'-dihydroxy semicarbazone as an analytical reagentInternational Journal of Pharma and Bio Sciences. 2011 Oct-Dec; 2(4):315-319.
7. Min Jung Jung, P. Venkateswaran, Young Seak Lee. Solvent extraction of nickel(II) ions from aqueous solutions using triethylamine as extractant. Journal of Industrial and Engineering Chemistry. 2008 January; 14(1):110-115.
8. C.F. Coleman. Amines as Extractants. Nucl. Sci. Eng.1963 October; 17(2):274-286.
9. C.F. Coleman, C.A. Blake Jr., K.B. Brown. Analytical potential of separations by liquid ion exchange.Talanta.1962 March; 9(3):297-323
10. V. Padmavathy, P. Vasudevan, S.C. Dhingra. Thermal and spectroscopic studies on sorption of nickel(II) ion on protonated baker's yeast. Chemosphere. 2003; 52:1807-1817.
11. D.K. Yadav, R. S. Lokhande, Shirish Pitale, S. P. Janwadkar, P. S. Navarkar and P. K. Rana. Solvent extraction and spectrophotometric determination of Mo(VI) by using acetophenone-2',4'-dihydroxy semicarbazone as an analytical reagent. International Journal of Pharma and Bio Sciences. 2012 Oct; 3(4):309-314.
12. Prabhakar S. Navarkar, Rama S. Lokhande, Shirish M. Pitale, Suhas P. Janwadkar, Paritosh K. Rana, Dilip K. Yadav. Liquid-liquid extraction and spectrophotometric determination of iron using acetophenone 2',5'-dihydroxy thiosemicarbazone (adhts) as a new analytical reagent. International Journal of Pharma Sciences and Research. 2012 Sep; 3(9): 459-462.

Reviewers of this article

Dr.Aniruddha Patil

Assistant professor in chemistry
Maharshi Davanand college of Arts Science and commerce
Dr.S.S.Road,Parel Mumbai - 400012

Prof.Dr. M.Ranga Priya, M.Pharm., Ph.D., R.Ph.

Professor , Dept of Pharmaceutics,Sun Institute Of Pharmaceutical Education & Research, Kakupall, Nellore Rural, Nellore, Andhra Pradesh 524346

Prof.Dr.K.Suriaprabha

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